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Advantages of flexible auto-dilution sample introduction solutions for ICP OES

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ABSTRACT

Purpose: To demonstrate the capabilities of auto-dilution sample introduction solutions, such as the Teledyne CETAC SDX HPLD and ESI prepFAST, when coupled to ICP-OES systems.

Methods: An ICP-OES system was used in conjunction with two different auto-dilution systems to carry out a complex analytical method with reduced time and user effort with respect to manual sample preparation.

Results: Auto-dilution systems like the ESI prepFAST and the Teledyne CETAC SDX HPLD provide similar accuracy and precision to manual dilutions while ensuring that the speed of the sample preparation process and method setup is not compromised.

INTRODUCTION

Fully automated sample introduction systems that include an autosampler equipped with an intelligent auto-dilution solution play an important role in routine laboratories to streamline workflows and improve productivity. Complete sample introduction systems like these provide a simple integrated workflow that is easily accessible to all users.

By fully automating both, prescriptive and intelligent auto-dilution steps, manual dilution during analyses is eliminated. This increases productivity, prevents post-analysis re-runs of samples and ultimately reduces cost of ownership. As an example, this paper describes the analysis of waste waters using Inductively Coupled Plasma Optical Emission Spectroscopy (ICP-OES) in conjunction with a fully automated, intelligent auto-dilution system.

A Thermo Scientific™ iCAP™ 7400 ICP-OES Duo equipped with a standard sample introduction kit was used with both an ESI prep*FAST* Auto-dilution system and a Teledyne CETAC SDX High Performance Liquid Dilution system to perform the analysis of waste waters according to US EPA Method 200.7[1]. The sample introduction kit components and the instrument parameters used are listed in Tables 1 and 4 for the CETAC SDX and ESI prepFAST setups respectively.

MATERIALS AND METHODS

Instrumentation

The iCAP 7400 ICP-OES Duo (Figure 1) was used in all experiments described in this poster. Method parameters, listed in Table 1 and 4 were applied for the according analyses. The duo configuration was chosen for its ability to detect trace elements in the axial view and for its wide dynamic range which can be extended with the radial view. Two different auto-dilution sample introduction systems were used (Teledyne CETAC SDX High Performance Liquid Dilution System (HPLD) (Figure 2) and ESI prep*FAST* (Figures 6 and 7)) to transfer the sample to the introduction system of the ICP-OES and to carry out automatic prescriptive and intelligent dilutions, respectively.

Figure 1. The Thermo Scientific iCAP 7400 Duo ICP-OES with both radial and axial viewing capabilities

Data Analysis

For each element, wavelengths were selected using the intuitive wavelength selection tool of the Thermo Scientific™ Qtegra™ Intelligent Scientific Data Solution™ (ISDS) Software. To ensure freedom from interferences, the subarray plots were examined and background correction points were set appropriately, as detailed in Table 3.

Sample preparation

All calibration and spike solutions were prepared from 1000 µg·g^{-1} or 10000 µg·g^{-1} single element stock solutions. The individual solutions were made up with 18 MΩ ultra-pure water and trace metal grade HNO_3 to a final concentration of 1% HNO_3 for each solution. To account for physical interferences due to differing matrix compositions, an internal standard solution of yttrium (10 µg·g^{-1}) and 5% CsCl was added online to all solutions either via a Y-piece or directly via the auto-dilution system.

A multi element stock calibration solution was prepared according to the concentrations shown in Table 2 and multi-point calibration points generated by dilution via the auto-dilution system.

CETAC SDX HPLD

Table 1. ICP-OES Method parameters using the Teledyne CETAC SDX HPLD Auto-dilution System.

Parameter	Setting	
Pump tubing	Sample Tygon™ white/white Drain Tygon™ blue/yellow Internal standard Tygon™ orange/green	
Pump speed	50 rpm	
Spray chamber	Glass cyclonic	
Nebulizer	Glass concentric	
Center tube	2 mm	
Torch	EMT	
Nebulizer gas flow	0.5 L·min^{-1}	
Auxiliary gas flow	0.5 L·min^{-1}	
Coolant gas flow	12 L·min^{-1}	
RF power	1150 W	
Wash time	30 s	
Exposure time	Axial view	Radial view
	UV 15 s, Vis 5 s	Vis 5 s

Figure 2. Teledyne CETAC SDX HPLD System

Table 2. Auto-dilution factors and concentrations of calibration standards in µg·g^{-1}.

Element	Stock Solution	STD 1	STD 2	STD 3	STD 4
Auto-dilution factor		100	10	5	1
Ag	50	0.5	5	10	50
Al	100	1	10	20	100
As	50	0.5	5	10	50
B	50	0.5	5	10	50
Ba	100	1	10	20	100
Cd	50	0.5	5	10	50
Co	50	0.5	5	10	50
Cr	50	0.5	5	10	50
Cu	50	0.5	5	10	50
Fe	100	1	10	20	100
Ni	50	0.5	5	10	50
P	500	5	50	100	500
Pb	50	0.5	5	10	50
Sb	50	0.5	5	10	50
Se	50	0.5	5	10	50
Sn	50	0.5	5	10	50
Ti	50	0.5	5	10	50
Tl	50	0.5	5	10	50
V	50	0.5	5	10	50
Zn	50	0.5	5	10	50

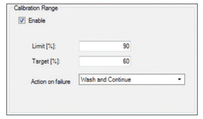
Table 3. Known and measured concentrations of the QCS and IPC as well as recoveries before and after spiking.

Element and wavelength (nm)	View	Internal standard wavelength (nm)	LDR (µg·g^{-1})	MDL (ng·g^{-1})	Required MDL (ng·g^{-1})
Ag 328.068	Axial	Y 377.433	> 10	0.7	4
Al 396.152	Radial		> 100	57	667
As 189.042	Axial	Y 224.306	> 50	0.3	7
B 208.959	Axial	Y 360.073	> 10	2.1	1000
Ba 455.403	Radial	Y 377.433	> 100	2.3	667
Cd 228.802	Axial	Y 224.306	> 10	1.1	3
Co 228.616	Axial	Y 360.073	> 50	0.6	23
Cr 284.325	Axial	Y 371.030	> 50	3.8	17
Cu 324.754	Axial	Y 224.306	> 50	3.2	33
Fe 259.940	Radial	-	> 100	16	1000
Ni 231.604	Axial	Y 224.306	> 10	0.9	17
P 177.495	Axial	Y 224.306	> 50	4.4	333
Pb 220.353	Axial	Y 377.433	> 10	1.9	17
Sb 206.833	Axial	Y 224.306	> 10	6.9	10
Se 196.090	Axial	Y 324.228	> 10	4.7	12
Sn 189.989	Axial	-	> 50	2.8	12
Ti 334.941	Axial	-	> 10	0.6	2
Tl 190.856	Axial	Y 224.306	> 10	4.4	17
V 309.311	Axial	Y 360.073	> 50	1.6	17
Zn 213.856	Radial	Y 224.306	> 50	4.8	67

To demonstrate the capability of the intelligent auto-dilution system for dilution of samples that are outside the calibration range, the reference material was spiked with 100 µg·g^{-1} of Cu and Fe. The parameters for intelligent dilution were set according to Figure 3.

The detected concentration was approximately two times above the upper calibration limit of copper and directly at the upper calibration limit of iron (see Figure 4).

Figure 3. Settings within Qtegra ISDS Software for intelligent dilution of samples outside the calibration range.



After analysis of the sample, Qtegra ISDS Software intelligently added a sample to the sample list with an automatically calculated auto-dilution factor such that the diluted concentration fell at the calibration graphs at 60% of the upper calibration limit for both elements (see Figure 5).

Figure 4. Original sample concentrations on the calibration graphs for Cu 324.754 and Fe 259.940.

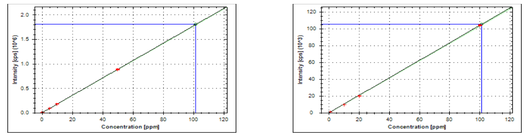
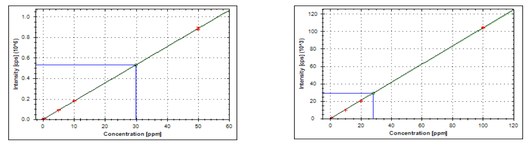


Figure 5. Sample concentration on the calibration graphs for Cu 324.754 and Fe 259.940 after intelligent auto-dilution with a calculated factor of 3.537 using the Teledyne CETAC SDX HPLD system and Qtegra ISDS Software.



ESI prep*FAST* Auto-dilution System

Figure 6. The ESI prep*FAST* Auto-dilution system for the iCAP 7400 ICP-OES.

Figure 7. Sample list within Qtegra ISDS Software showing ESI *prep*FAST dilution factors for the calibration standards and one intelligently diluted and automatically inserted sample.

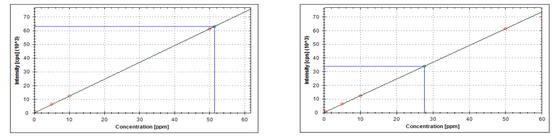
	Label	Status	Repeats	Sample Type	prepFAST DF
1	Blank		3	BLK	1
2	STD 1		3	STD	100
3	STD 2		3	STD	10
4	STD 3		3	STD	5
5	STD 4		3	STD	1
6	Sample		3	UNKNOWN	1
7	Sample		3	UNKNOWN	1.715

Table 4. ICP-OES method parameters using the ESI *prep*Fast Auto-dilution system.

Parameter	Setting	
Pump tubing	Sample Tygon™ white/white Drain Tygon™ blue/yellow	
Pump speed	50 rpm	
Spray chamber	Glass cyclonic	
Nebulizer	Glass concentric	
Center tube	2 mm	
Torch	EMT	
Nebulizer gas flow	0.5 L·min^{-1}	
Auxiliary gas flow	0.5 L·min^{-1}	
Coolant gas flow	12 L·min^{-1}	
RF Power	1150 W	
Wash time	20 s	
Exposure time	Axial view	Radial view
	UV 15 s, Vis 5 s	Vis 5 s

As with the Teledyne CETAC SDX HPLD, the ESI *prep*FAST is able to accurately generate calibration curves from single stock solutions. Qtegra ISDS Software is also able to use the system to automatically and intelligently dilute over-range samples to generate valid data with minimal user intervention, as demonstrated in Figure 8.

Figure 8. Sample concentrations on the calibration graph for Tl 190.856 before (left) and after (right) intelligent auto-dilution with a calculated factor of 1.715 using the ESI *prep*FAST system and Qtegra ISDS Software.



RESULTS

The study shows that the Thermo Scientific iCAP 7000 Plus Series ICP-OES in conjunction with auto-dilution systems delivers very good accuracy and sensitivity for the analysis of elements in conformity with 40 CFR Part 437 using EPA method 200.7. With the CETAC SDX HPLD system the total analysis time of a sample depends on the dilution factor whereas with the ESI prepFAST the measurement time is the same for each sample, details are shown in Table 5.

Table 5. Analysis time including uptake and wash time with CETAC SDX HPLD and ESI prepFAST.

	CETAC SDX HPLD	ESI prepFAST
Measurement time 1 x dilution	2 min 50 sec	2 min 30 sec
Measurement time 5 x dilution	3 min 37 sec	2 min 30 sec
Measurement time 10 x dilution	3 min 33 sec	2 min 30 sec
Measurement time 100 x dilution	3 min 29 sec	2 min 30 sec

CONCLUSIONS

Automation of laboratory workflows has taken a step forward with the full integration of auto-dilution. The ESI prep*FAST* and the Teledyne CETAC SDX Auto-dilution systems have been demonstrated to be powerful, flexible and robust tools in routine trace elemental analyses by the iCAP 7000 Series ICP-OES. Control of these complete systems with Thermo Scientific Qtegra ISDS Software provides a single, intuitive and fully integrated workflow, eliminating manual dilution in both prescriptive and intelligent forms, and realizing fully automated analyses.

With auto-dilution systems, user interaction and the possibility for human error are greatly reduced. Auto-dilution comprises three simple steps from stock solution to results, whereas manual dilutions need multiple preparation steps, manual review of acquired data and time-consuming manual re-dilutions where necessary. Eliminating manual intervention increases productivity, prevents re-runs and ultimately reduces cost of ownership.

REFERENCES

1. Application Note 43376 - U. S. EPA Method 200.7 - Wastewater Analysis for Trace Metals Using an Auto-Dilution System Coupled to the Thermo Scientific iCAP 7000 Plus Series ICP-OES.

TRADEMARKS/LICENSING

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